

DEVELOPMENT AND CHARACTERISATION OF TREATED KAOLIN FILLED
POLYPROPYLENE/KAOLIN NANOCOMPOSITES

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DEDICATION

Specially dedicated to Allah Subhaana Wa Ta'ala (SWT) that gave me the strength to undertake this research work and my late parents (mother and father; Malama Salimatu and Mallam Yahaya); May Allah (SWT) rest their souls in perfect peace. Ameen.



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ABSTRACT

This research work focused on producing modified kaolin filler in polypropylene/kaolin (PP/K) nanocomposite by melt compounding process in order to improve its mechanical and thermal properties for industrial applications. The surface treatments of micron sized Malaysian kaolin were conducted to produce nano sized kaolin by acidification of kaolin fillers with sulphuric acid and planetary milling using urea (mechanochemical milling). Testing on both surface treated kaolin were carried out with the aid of Field Scanning Electron Microscopy (FESEM), Fourier Transform Infrared (FTIR), Brunauer–Emmett–Teller (BET), X-ray Diffraction (XRD) and Particle Size Analyser and results of both treated kaolin were compared. However, the surface treated kaolin using acidification was unsuccessful as shown by XRD, FTIR and BET results. A successful delamination of micron sized into nano sized kaolin was achieved by mechanochemical milling. The additional bands at 3624, 3445 and 3388 cm^{-1} and illite phase at lower 2θ by FTIR and XRD studies respectively, indicated delamination of kaolin. Surface area increased by 400% from BET results. The PP/K nanocomposite was produced by incorporating low weight (1-7%) percentages of organically modified nanokaolin into PP by melt compounding with polypropylene grafted maleic anhydride (PP-g-MA) as coupling agent. The FTIR and XRD analyses on chemical structure showed successful synthesis of PP/K nanocomposites by the vanishing of characteristic of OH bands and peaks of kaolin respectively. The tensile and impact strength, $\tan \delta$, loss modulus and melt flow index of PP/K nanocomposite decreases by 17, 27, 36, 32 and 78% respectively. Conversely, the results show that incorporation of nanokaolin clay into PP causes increase in thermal degradation (200%), crystallinity (17%), nucleation effect (17%), storage modulus (10%), surface roughness (87%), and optical (262%). Whereas, TEM of PP/K nanocomposite exhibit nanokaolin dispersion with nanoscale sizes. Therefore, the PP/K nanocomposites formulated shall be a potential candidate for manufacturing novel new materials of attraction in many sectors.

TABLE OF CONTENTS

TITTLE	i
DECLARATION	ii
DEDICATION	iii
ACKNOWLEDGEMENTS	iv
ABSTRACT	v
ABSTRAK	xi
TABLE OF CONTENTS	vi
LIST OF FIGURES	xi
LIST OF TABLES	xv
LIST OF APPENDICES	xvi
LIST OF SYMBOLS AND ABBREVIATIONS	xvii
CHAPTER 1 INTRODUCTION	1
1.1 Research background	1
1.2 Problem statement	6
1.3 Research objectives	8
1.4 Research scope	9
1.5 Significance of the study	10
CHAPTER 2 LITERATURE REVIEW	11
2.1 Introduction	11
2.2 Clay mineral fillers for polymer nanocomposites	13
2.2.1 Kaolin clay	14
2.2.2 Elemental analysis of Malaysian kaolin	17
2.3 Surface treatments of kaolin	18
2.3.1 Kaolin surface treatments with acids	22
2.3.2 Kaolin surface treatments using urea	23
2.4 Polymer matrix nanocomposites (PMCs)	25

2.4.1	Polymeric matrices	31
2.4.2	Reinforcements	33
2.4.3	Additives	34
2.5	Polypropylene (PP) and its nanocomposites	36
2.6	Polypropylene/kaolin (PP/K) nanocomposites	39
2.7	Properties and characterizations of PP/K nanocomposite	42
2.8	Mechanical properties of PP/K nanocomposites	44
2.8.1	Tensile strength	45
2.8.2	Charpy impact strength	47
2.8.3	Dynamic mechanical analysis (DMA) in nanocomposites	49
2.9	Melt flow properties of PP/K nanocomposite	50
2.10	Optical properties of PP/K nanocomposite	52
2.11	Thermal properties of PP/K nanocomposite	54
2.11.1	Differential scanning calorimetry (DSC) in PP nanocomposites	55
2.11.2	Thermal gravimetric analysis	57
2.12	Morphological properties of PP/K nanocomposite	58
2.12.1	Scanning electron microscopy	58
2.12.2	Atomic force microscopy	60
2.12.3	Transmission electron microscopy	62
2.13	Chemical structure analyses of PP/K nanocomposite	63
2.13.1	Fourier transform infrared spectroscopy	63
2.13.2	X-ray powder diffraction	64
2.14	Applications of PP/K nanocomposites	66
CHAPTER 3	METHODOLOGY	69
3.1	Introduction	69
3.2	Materials	70
3.3	Kaolin surface treatment with sulphuric acid	71
3.4	Kaolin surface treatment with urea using mechanochemical milling	72
3.5	Sample preparation of Polypropylene/kaolin (PP/K) nanocomposites	73

3.6	Samples characterisations	76
3.6.1	Elemental analysis	76
3.6.2	Particle size analysis	76
3.6.3	Surface area analysis	77
3.6.4	Mechanical properties of PP/K nanocomposites	77
3.6.4.1	Tensile strength (MS ISO 527)	77
3.6.4.2	Impact test (MS ISO 179)	78
3.6.5	Melt flow properties of PP/K nanocomposite	79
3.6.6	Optical properties PP/K nanocomposite	80
3.6.7	Thermal properties of PP/K nanocomposite	80
3.6.7.1	Differential scanning calorimetry	81
3.6.7.2	Thermogravimetry analysis	82
3.6.8	Morphological analyses	83
3.6.8.1	Field scanning electron microscopy (FESEM)	83
3.6.8.2	Atomic force microscopy (AFM)	85
3.6.8.3	Transmission electron microscopy (TEM)	85
3.6.9	Chemical structure analyses	86
3.6.9.1	Fourier transform infrared (FTIR)	86
3.6.9.2	X-ray powder diffraction (XRD)	87
3.6.10	Dynamic mechanical analysis (DMA)	89

CHAPTER 4 RESULTS AND DISCUSSION **91**

4.1	Introduction	91
4.2	Kaolin surface treatment with sulphuric acid	91
4.2.1	Particle size analysis	92
4.2.2	Chemical structure analyses	98
4.2.2.1	Fourier transform infrared spectroscopy	98
4.2.2.2	X-ray diffraction (XRD)	99
4.3	Kaolin surface treatment with urea using the mechano-chemical method	101
4.3.1	Particle size analysis	101
4.3.2	Surface area analysis	104
4.3.3	Morphological analysis	107

4.3.4	Chemical structure analyses	110
4.3.4.1	Fourier transform infrared spectroscopy	110
4.3.4.2	X-ray diffraction	112
4.4	Elemental analyses of kaolin before and after treatment with H ₂ SO ₄ acid and urea	114
4.5	Polypropylene/kaolin (PP/K) nanocomposites	118
4.6	Mechanical properties of PP/K nanocomposites	119
4.6.1	Tensile strength of PP/K nanocomposites	119
4.6.2	Charpy impact strength	122
4.6.3	Dynamic mechanical analysis (DMA)	124
4.7	Flow properties of PP/K nanocomposites	128
4.7.1	Effect of kaolin loading	128
4.7.2	Effect of temperature	129
4.7.3	Effect of pressure	131
4.8	Optical properties of PP/K nanocomposite	131
4.9	Thermal properties of PP/K nanocomposites	134
4.9.1	Crystallization behaviour	134
4.9.2	Nucleation activity	139
4.9.3	Crystallization activation energy	140
4.10	Thermal degradation of PP/K nanocomposites	142
4.10.1	Effect of kaolin loading	142
4.10.2	Effect of temperature	144
4.10.3	Effect of pressure	145
4.11	Morphological properties of PP/K nanocomposites	146
4.11.1	Field scanning electron microscopy (FESEM)	146
4.11.2	Atomic force microscopy (AFM)	149
4.11.3	Transmission electron microscopy (TEM)	151
4.12	Chemical structure analyses of PP/K nanocomposite	154
4.12.1	Fourier transform infrared spectroscopy (FTIR)	155
4.12.2	X-ray powder diffraction	156
CHAPTER 5	CONCLUSION AND RECOMMENDATIONS	159
5.1	Conclusion	159
5.2	Recommendations	161

REFERENCES**162****APPENDICES****192****VITA**

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ABSTRAK

Penyelidikan ini tertumpu kepada penghasilan pengisi kaolin yang diubahsuai dalam nanokomposit polipropilena/kaolin (PP/K) secara penyebatian leburan untuk meningkatkan sifat mekanikal dan termalnya bagi aplikasi perindustrian. Rawatan permukaan telah dilakukan pada kaolin bersaiz mikron bagi menghasilkan kaolin bersaiz nano menerusi pengasidan pengisi kaolin dengan asid sulfurik, dan pengisaran planet dengan urea (pengisaran mekanokimia). Kedua-dua kaolin terawat permukaan telah diuji dengan Mikroskop Elektron Pengimbasan Medan (FESEM), Infra Merah Penjelmaan Fourier (FTIR), Brunauer-Emmett-Teller (BET) serta Pembelauan Sinar-X (XRD), dan keputusan ujian kedua-dua kaolin tersebut telah dibandingkan. Kaolin terawat secara pengasidan adalah tidak berhasil seperti yang ditunjukkan oleh ujian XRD, FTIR dan BET. Pelekangan mikron bersaiz besar ke dalam kaolin bersaiz nano telah dicapai dengan pengisaran mekanokimia. Jalur tambahan pada 3624, 3445 dan 3388 cm^{-1} dan fasa illit pada sudut 2θ yang kecil menerusi FTIR dan XRD masing-masing, menunjukkan pelekangan kaolin. Kawasan permukaan meningkat 400% menerusi ujian BET. Nanokomposit PP/K dihasilkan dengan menggabungkan peratusan nanokaolin organik yang rendah secara organik (1-7%) dan polipropilena terubahsuai maliek anhidrida (PP-g-MA) sebagai agen pengganding dengan meleburkan penyebatian. Analisis kimia menerusi FTIR dan XRD menunjukkan kejayaan sintesis nanokomposit PP/K apabila ciri-ciri jalur OH dan puncak kaolin masing-masing tidak dapat dikesan. Kekuatan tegangan dan hentaman, $\tan \delta$, modulus kehilangan dan indeks aliran leburan nanokomposit PP/K masing-masing berkurang 17, 27, 36, 32 dan 78%. Penambahan tanah liat nanokaolin ke dalam PP telah meningkatkan degradasi haba (200%), kehabluran (17%), kesan penukleusan (17%), modulus penyimpanan (10%), kekasaran permukaan (87%), dan optik (262%). Ujian TEM terhadap nanokomposit PP/K mempamerkan serakan kaolin bersaiz nano. Oleh itu, nanokomposit PP/K ini berpotensi dalam penghasilan bahan novel menarik di pelbagai sektor.

LIST OF FIGURES

1.1	Map of Malaysia showing the location industrial mineral reserves of peninsular Malaysia	8
2.1	Structure of kaolin	15
2.2	Structure of layered silicate of type 2:1	15
2.3	Schematic illustration of a representative XRF system	17
2.4	Benzamide	21
2.5	Ethyl pyridinium chloride	21
2.6	Mechanism of intercalation of urea and kaolin	25
2.7	Monomer of polypropylene	37
2.8	Molecules of PP polymer in isotactic, syntactic and atactic forms	38
2.9	Structure of three major types of layered silicates in polymer matrix	39
2.10	Tensile test machine (Maiti and Lopez, 2005)	46
2.11	Impact test machine (Hussain <i>et al.</i> , 2006)	48
2.12	General Schematic of DMA instrument	50
2.13	Outline of the dimension of melt flow index	51
2.14	Schematic diagram of UV-Spectrometer	54
2.15	Schematic diagram of a differential scanning calorimeter	56
2.16	The schematic diagram of the thermogravimetric analyzer configuration	57
2.17	Scanning Electron Microscopy	59
2.18	Schematic illustration of AFM	61
2.19	Schematic illustration of the TEM	62
2.20	Schematic illustration of classic FTIR spectrometer	64
2.21	Schematic illustration of 2D-XRD	65
3.1	Research framework	70
3.2	Fritch Pulverisette 6 planetary milling machine	72
3.3	Elmasonic E30H ultrasonic bath	73
3.4	PW 3000 two-roll mill	74

3.5	NP7- 1F injection moulding machine	74
3.6	Images representing samples for (a) Tensile test and (b) Impact test	75
3.7	Lloyd instrument tensile strength machine	78
3.8	Zwick/ Roell 4106 instrument melt flow index machine	80
3.9	Universal V4.5A TA DSC instrument	82
3.10	Result data of thermogravimetry analysis	83
3.11	JEOL JSM-7600F FESEM machine	84
3.12	JEOL-H-800 TEM machine	86
3.13	Perkin Elmer Spectrum 100	87
3.14	XRD Model Bruker D8 Advance	89
4.1	Particle size distribution of untreated and treated kaolin at different H_2SO_4 of concentrations	93
4.2	FESEM micrographs observed at magnification of $5,000\times$ for untreated and treated kaolin at different H_2SO_4 concentrations	95
4.3	FESEM micrographs observed at magnification of $500\times$ and EDX analyses of before and after kaolin treatment with different H_2SO_4 concentrations	97
4.4	FTIR of untreated and treated kaolin at different H_2SO_4 concentrations	99
4.5	XRD of untreated and treated kaolin at different H_2SO_4 concentrations	101
4.6	Particle size distribution of (a) untreated kaolin, and (b) treated kaolin using urea	102
4.7	N_2 adsorption/desorption isotherms of (a) untreated and (b) treated kaolin	105
4.8	Pore size distribution of untreated kaolin (UK)	107
4.9	Pore size distribution of treated kaolin (TK)	107
4.10	FESEM micrographs displaying the morphological alterations of the kaolin crystals as a result of mechano-chemical milling	108
4.11	SEM-EDX analyses of (a) untreated and (b) treated kaolin with urea	110
4.12	FTIR of urea, untreated and treated kaolin samples	111
4.13	XRD patterns of untreated and delaminated kaolin	114
4.14	Tensile strength of PP/K nanocomposites at different kaolin loadings	120
4.15	Impact strength of PP/K nanocomposites at different kaolin loading	122
4.16	FESEM micrographs displaying the fracture part of impact test	124
4.17	DMA spectra of PP and PP/K nanocomposites	127

4.18	The MFI values of PP/K nanocomposites obtained at different kaolin loadings	129
4.19	The MFI curves of PP/K nanocomposites obtained at different processing temperature	130
4.20	The MFI values of PP/K nanocomposites at different pressure	131
4.21	Reflectance spectra of pure PP and various PP/K nanocomposites	133
4.22	DSC crystallisation curves of PP and PP/K nanocomposites with different nanokaolin loadings	135
4.23	DSC melting curves of PP and PP/K nanocomposites with different nanokaolin loadings	136
4.24	Non-isothermal crystallisation curves of PP and PP/K nanocomposites	137
4.25	Effective activation energy of PP and PP/K nanocomposites	141
4.26	Kissinger plot for determining non-isothermal crystallization activation energy of PP with variation of nanokaolin content	141
4.27	TGA curves of netPP and PP/K nanocomposites	144
4.28	FESEM micrographs of (a) Pure PP, (b) PPNK5, (c) PPK5 and (d) PPK7 (magnification of 5,000×)	147
4.29	FESEM fractographs for samples of (a) pure PP, (b) PPK1, (c) PPK3, (d) PPK5 and (e) PPNK5 (magnification of 1,000×)	148
4.30	AFM 2D and 3D topographic images of (a) PP (b) PPK1 and (c) PPK3 (d) PPK5 and (e) PPK7	150
4.31	TEM micrograph for PPK5 nanocomposites with 5 wt% of nanokaolin	152
4.32	TEM micrograph for PPK3 nanocomposite with 3 wt% of nanokaolin	153
4.33	TEM micrograph for PPK1 nanocomposite with 1 wt% of nanokaolin	154
4.34	FT-IR spectra of pure PP, PPK1, PPK3 and PPK7	156
4.35	XRD spectra of PP and PP/K nanocomposites	158

LIST OF TABLES

2.1	Surface reformations of kaolin	19
2.2	Commonly used operational additives for PP matrix.	36
2.3	Commonly used characterisation techniques and their properties (Zeng <i>et al.</i> , 2005).	44
2.4	Types of thermal analysis	55
3.1	Formulation of blends of the PP/K nanocomposites samples	75
4.1	Particle size and surface area analyses (SSA) of kaolin samples	93
4.2	The corresponding wavenumber of silica	98
4.3	Particle size analyses of untreated and treated kaolin with urea	103
4.4	Surface properties of untreated and treated kaolin samples	106
4.5	EDX of kaolin samples before and after urea treatment	109
4.6	Mineralogical compositions of kaolin samples treated with H ₂ SO ₄ acid	114
4.7	Mineralogical composition of kaolin samples treated with urea	117
4.8	Comparison between surface treated kaolin particles using H ₂ SO ₄ acid and urea	118
4.9	The DSC results of PP and PP/K nanocomposites	140
4.10	Crystallization activation energy change ΔE and crystallization half-time ($t_{0.5}$) of the samples	142
4.11	Thermal stability of pure PP and PP/K nanocomposites	143
4.12	TGA data of PP and PPK5 attained in atmosphere (oxidative)	145
4.13	Surface roughness of PP and PP/K nanocomposites	151
4.14	Assignment of principle bands of kaolin and PP	155

LIST OF APPENDICES

A	List of publications	192
B	Data sheet polypropylene	200
C	Data sheet kaolin	202
D	Data sheet polypropylene grafted malaiec anhydride	204
E	Data sheet sulphuric acid	206
F	Data sheet urea	217



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LIST OF SYMBOLS AND ABBREVIATIONS

AFM	Atomic force microscopy
ASTM	American Standards Test Method
BET	Brunauer-Emmet-Teller
cm ²	Square centimeter
cm ³	Centimeter cubic
°C/min	Degree celsius per minute
<i>d</i>	Interspacing between diffraction lattice plane
DMA	Dynamic mechanical analysis
DSC	Differential scanning calorimetry
EDX	Energy dispersive X-ray
FESEM	Field emission scanning electron microscopy
FTIR	Fourier transform infra-red
g	Gram
GPa	Giga pascal
H _f	Heat of Fusion
HRTEM	High resolution transmission electron microscopy
K	Kaolin
MFI	Melt flow index
mg	Milligram
PP	Polypropylene
PP-g-MA	Polypropylene grafted maleic anhydride
PP/K	Polypropylene/kaolin
PMCS	Polymer matrix composites
PNC	Polymer nanocomposites
SAXS	Small-angle X-ray scattering
SSA	Specific surface area
T _c	Crystallization temperature

TEM	Transmission electron microscopy
T_g	Glass transition temperature
TGA	Thermogravimetric analysis
WAXRD	Wide angle X-ray diffraction
wt%	Weight percent
X_c	Crystallinity content
XRD	X-ray diffraction
α	Alpha
θ	Diffraction angle
λ	Wavelength
μm	Micron
ΔH_{100}	Theoretically heat of fusion of 100% crystalline polymer



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CHAPTER 1

INTRODUCTION

1.1 Research background

Demands for high performance materials have been increasing day-in, day-out, hence the focus of current research has been manufacturing products with superior properties at slight deviations in the tools, procedures and cost of operations (Krishnan *et al.*, 2013). Surface treatments are more precisely on surface engineering or alteration of surfaces in engineering to the definite anticipated properties. It leads to enhancement in surface area, regulation of friction and wear, advance corrosion opposition, alterations in physical properties (for example reflection, resistivity and conductivity), modification of the material dimension, adaptability of appearance (such as roughness and color) and reduce cost (Lee, 2000). These could be achieved through acidification (refluxing), ion-exchange method (Shah *et al.*, 2016), mechanical comminution (Wang *et al.*, 2012) and grafting-intercalation using dry-grinding method (Makó *et al.*, 2009).

Nanocomposites have an annual growth rate of approximately 25% and they are the largest group to be in engineering plastics and elastomers (Henrique *et al.*, 2009). Once, the levels of technology turn out to be more sophisticated, the materials used are expected to be satisfyingly more efficient. Several performance features are expected from these materials. For instance, the materials for high-end applications, such as aerospace engineering should have higher performance efficiency and reliability if compared to that of other engineering applications. The materials have to be light weight for many applications including thermoplastic storage compartments used in airplanes. The resulting products have to be energy efficient

and cost effective (Sinha and Okamoto, 2003). In the automotive industries, constituents for the under hood, exterior and interior, are progressively substituting metal components by reinforced composites owing to decrease in their weights, better-quality functioning, additional beautiful look and consistency. In the present development polypropylene (PP) is the greatest extensively consumed polyolefin which more than half of the entire plastic materials are accountably are used in automobiles (Hasegawa *et al.*, 1998). In addition, the materials used must have good blend of properties, such as enhanced tensile and flexural strength. To sum up, these products of modern technological source may also need to function in environments that are extreme, such as, very high temperature (the order of 2500K), cryogenic conditions, highly corrosive, vacuum (as in space), high hydrostatic pressure (as in deep sea), high electric, and magnetic or irradiation fields (Mittal, 2009).

The conventional materials may not always be capable of meeting the demands of such above-mentioned extreme environments. New materials are being created for meeting these performance requirements. Thus, polymer composite materials had been developed as one class of such material system. As a challenge to accomplish these objectives, researchers are exploring several polymer nanocomposites with diverse fillers and additives (Hegde, 2009); including evolution from microfiller to nanofiller particles which produces dramatic changes in physical and chemical properties of such materials. The materials that are in nanoscale have larger surface area for a given volume (Luo and Daniel, 2003) than conventional ones (micron sized materials). A nanostructured material can have substantially different properties from larger-dimensional material of the same composition.

Polymer nanocomposites are polymers (thermoplastics, thermosets or elastomers) which have been reinforced with small amount ($< 5\%$ by weight) of fillers of very high aspect ratio ($AR > 300$). The aspect ratio of a geometric shape is the ratio of its sizes in different dimensions. (Throne, 2005). High aspect ratio nanoparticles (HARN) are nanoparticles with a length many times that of their width. In previous decades, polymer nanocomposites have risen as a novel class of materials and fascinated substantial interest in research and development activities universally (Zeng *et al.*, 2005). Polymer nanocomposites are still one of the dynamically development materials and has a long history. The production and characterisation of new and novel materials are part of the leading objectives of advanced material research. The task to yield stronger, tougher, light-weight

materials continues rapidly, being determined by demands for property enhancements, economy and material obtainability (Browska, 2013). In order to improve polymer properties, the introduction of small amount (up to 5% wt.) of inorganic nanofillers in polymer matrices is a motivating technique. Among the roles played by low amount of fillers is that density of final product are reduced; leading to advanced fuel efficiency and reduced pollution. It is obvious that nanocomposites offer similar or improved properties at considerable lower filler loading levels than materials with conventional fillers.

Addition of nanofillers to polymers allows us possibly to produce composite materials with upgraded mechanical and barrier properties, flame retardancy, electrical conductivity. This is mainly due to their new and frequently much enhanced mechanical, thermal, electrical and optical properties as compared to their macro- and micro-counterparts. The aim of the fabrication of thermoplastic composites is mostly directed by price performance relationship. Mineral fillers have made a significant input to the remarkable growth of thermoplastic polymers composites (Meziane *et al.*, 2016). This emphasis on filled polymer composites is due to a combination of factors. In addition to reduction of price of the final material, mineral fillers can equally help to increase shrinkage on moulding, stiffness and flammability, which are the major limitations of the bulk thermo-plastics. The mechanical and other properties of composites were affected by fillers depending on their shape, particle and aggregate sizes, surface characteristics, and degree of dispersion (Guessoum *et al.*, 2012).

Precisely, polymer nanocomposites prepared by using layered inorganic substrates display high heat distortion temperatures, improved flame resistance, better modulus values, enhanced barrier properties and reduced thermal expansion coefficient at low cost (Gilman *et al.*, 2000). The synergistic effects of the nano scale structure and particle–particle interactions are likely responsible for these improved properties protracted to the polymer surfaces. The profound understanding of polymer nanocomposites was achieved by developments in microscopy: scanning tunnelling microscopy, scanning electron microscopy and transmission electron microscopy. By means of these authoritative characterisation tools, scientists can understand better and account for surface topology, structure and morphology up to atomic scale (Hegde, 2009). Consequently, the nanocomposite technology has

initiated novel engineering materials with an efficient and countless strategy in upgrading the new structural and functional properties of synthetic polymers.

The types of polymers used in preparation of polymer nanocomposites include:

- i. Vinyl polymers which consist of the vinyl addition polymers derivative of conventional monomers such as acrylic acid (Sinha and Okamoto, 2003), acrylonitrile, methyl methacrylate (Blumstein and Malhotra, 1970), methyl methacrylate copolymers, other acrylates and styrene.
- ii. Condensation (step) polymers; numerous mechanically significant polycondensates have been utilised in nanocomposites formulation through layered silicate. These comprise of poly(1-caprolactone) (PCL), N6 (Parfitt and Greenland, 1970; Lindström *et al.*, 2016), several other polyamides and polybutadiene.
- iii. Polyolefins for instance polypropylene (PP) (Kaempfer *et al.*, 2002), polyethylene oligomers, poly(ethylene-covinyl acetate) (EVA) polyethylene (PE) (Rong *et al.*, 2001) have been used.
- iv. Specialty polymers adding to the above stated common involving polymeric materials that would not include the use of poisonous or harmful constituents in their production, and might permit degradation through an environmental composting course. Consequently, polylactide (PLA) is of growing marketable attention since it is prepared from renewable sources and voluntarily biodegradable (Henrique *et al.*, 2009). Other biodegradable polymers for preparation of nanocomposites are unsaturated polyester, poly(butylene succinate) (PBS), aliphatic polyester (Maiti *et al.*, 2007) and polyhydroxy butyrate.

Polypropylene (PP) compatibility with fillers has been improved by matrix modification by grafting it with reactive moieties, such as acrylic acid, acrylic esters, and maleic anhydride (Lee *et al.*, 2008). Obviously, there is a countless incentive to use completely the commercially and low-cost essential thermoplastic polymers, however, in several terms, the major markets are in PVC and polyolefin for as many applications as possible (Meziane *et al.*, 2016). PP is one of the most adaptable product thermoplastics with heat resistance, unlimited crystallinity, special moisture barrier, routine optical properties, low cost, high thermal stability and low density with high processability and recyclability. Conventional fillers include silicates, talc,

calcium carbonate and fibers. Out of the inorganic fillers that can be incorporated into PP to improve its properties; clays are recognised to acquire several advantageous differences on stiffness, hardness, toughness and heat resistance (Lee *et al.*, 2005).

Kaolin is very much in abundance in Malaysia clayey soil, which can be made to reinforce PP to improve its properties at low cost of manufacture. The name kaolin perhaps originated from the Chinese term kauling, denotation for high ridge, the title of a hill near Jauchau Fu, China, somewhere the material was excavated centuries ago (Kotal and Bhowmick, 2015). It has fascinated additional and extra attention for its plentiful, low cost, and shielding effect in polymer. Kaolin are used in making tires, solid rubber tires, splash guards/wheel covers, seals/gaskets/o-rings, insulators, hose/belts, white roofing membranes, wire/cable, flooring, medical rubber goods, footwear and printing. Even though the chemical composition of kaolin is alike to halloysites, it differs by having a predominantly plate-like structure (Tang *et al.*, 2017).

Kaolin ($\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$) represents a 1:1 phyllosilicate with a gibbsite octahedral layer and a silicon oxide tetrahedral sheet. It is an asymmetric structure which allows the creation of hydrogen bonds among consecutive layers, developing a great cohesive energy. As the inorganic surface of the particles is tremendously polar while the PP is nonpolar, the interface interaction can be produced only by molecular modification of one or both components (Teng *et al.*, 2015). Therefore, to cultivate a good dispersion, the surface reformation of kaolin can be achieved either by the means of coupling agents, or through the intercalation of chemical species in the interlayer gallery, hence increase the basal spacing of the clay which may affluence the intercalation of polymer chains between its plate-like structures (Guessoum *et al.*, 2012).

Since the inorganic surface of the clay flakes is extremely polar whereas the PP is apolar, the boundary interface can be offered only by molecular reformation of one or both constituents (Guessoum *et al.*, 2012). Therefore, for a good dispersion, the surface treatment of kaolin can be accomplished either by the employment of coupling agents, otherwise by the intercalation of chemical groups in the interlayer passageway, to enlarge the basal spacing of the clay that will ease the intercalation of polymer chains among its clay particles (Frost *et al.*, 2002). Similarly, some of the chemical groups which can be directly inserted in the interlayer of the kaolinite are limited as a result of great cohesive energy between the clay layers. Horváth *et al.*,

(2005) stated that urea is one of the highly recognised compounds that can absolutely intercalate into kaolinite layers. Additional compounds that can also easily integrate into the kaolin flakes are dimethylsulphoxide (DMSO), potassium acetate, formamide and hydrazine (Valášková *et al.*, 2011).

In addition, the incompatibility held between the hydrophilic clays and non-polar hydrophobic PP inhibits suitable clay dispersal (Lertwimolnun and Vergnes, 2005). Accordingly, excessive attempts have been formulated to utilise the operational polyolefin oligomers as compatibilisers, adapted by any hydroxyl groups (OH) or maleic anhydride (MA) in form of polypropylene-grafted-maleic anhydride (PP-g-MA). These modifiers contain a definite amount of polar groups (i.e. $-\text{COOH}$, $-\text{OH}$) to incorporate between silicate sheets via hydrogen or additional chemical bond involved negative charges or in the oxygen group of the silicate plates (Dong and Bhattacharyya, 2008). Obviously, clay agglomerates are simply fragmented up and even dispersal of clay flakes can be prominently accomplished in the existence of PP-g-MA (Zdiri *et al.*, 2017).

The goal of this study is to synthesize PP nanocomposite by melt compounding; to improve its thermal and mechanical properties using organoclay (urea-kaolin intercalates) and PP as matrix with polypropylene maleic anhydride (PP-g-MA) as compatibiliser; then characterise their dispersion and morphology. The result is expected to make a great impact in the Malaysia economy and business and world at large.

1.2 Problem statement

The processing of polymer silicates nanocomposites to meet up with increasing mandate for high performance materials has been challenging. Kaolin is hydrophobic and polymers are hydrophilic (e.g. PP); obtaining uniform dispersion of fillers into the matrix through intercalation, porous assembly and surface loading tends to be difficult. Thus challenges in technological development of transition from micro to nanoparticles arises in today's research (Lv *et al.*, 2008). Modification of kaolin surface is therefore necessary. As a results, splitting along the (d_{001}) planes, cracking in platelets and rise in surface area occurs When fillers are added to a polymer matrix, will develop several properties of the composite material though it could have

a negative effect on a few other properties such as optical properties. Hence, it is of an excessive significance to have a balance among different properties; such as upgrading the properties of polymers; thermally and mechanically with increase barrier of PP.

Recently, mineral based functional composites has been successfully technologically advanced through compounding of a functional matrix (such as polypropylene, polyaniline and polyvinylchloride) and silicate materials (such as kaolin, bentonite and montmorillonite) at micro-nano level, by use of techniques such as intercalation, porous assembly and surface loading. These has been challenging in today's research. Classically, nanomaterials currently under investigation include nanoparticles, nanofibers, nanotubes, fullerenes, and nanowires. However, transition from micro to nanoparticles has yielded dramatic changes in physical properties of materials. Large surface area is exhibited for a given volume by nanoparticles because of their high aspect ratios (Luo and Daniel, 2003). The properties of a nanocomposite are seriously subjective to the size scale of its component phases and the grade of mixing between the two phases

Kaolin is very much underutilized raw materials in Malaysia; very few exploration of cheap kaolin is done for raw materials in industrial development. According to Department of Mineral and Geoscience, Malaysia; sizeable reserves of kaolin are found throughout Malaysia (Pui-Kwan, 2013). According to Joint Meteorological Group (JMG), the country has some 112 million tonnes of kaolin reserves located in the states of Perak, Johor, Kelantan, Selangor, Pahang and Sarawak as represented in Figure 1.1. There are 17 active kaolin mines operating mostly located in Bidor and Tapah district in Perak (Aw, 1986). Production of kaolin in 2010 was 530,331 tonnes value at RM 41,670,793 while 2015 was 261,574 tonnes value at RM 24.85 million (Minerals and Geoscience Department Malaysia, 2010).

The study uses Malaysian clay mined in Bidor, in the state of Perak, by KM company. It is located about 145 km north of Kuala Lumpur and 70 km south of Ipoh. Kaolin manufacture in the area was initiated on a small scale in the early 1930s, and currently accounts for more than two-thirds of Malaysia's kaolin output of about 50,000 tonnes per annum. Both wet and dry processes are used to produce various grades of kaolin for ceramics, paper, paint, plastics and rubber productions. The most developed kaolin manufactured is of paper-filler grade (Luo and Daniel, 2003).

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